1314

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE

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AN INVESTIGATION OF THE HIGH-TEMPERATURE PROPERTIES OF CHROMIUM-BASE ALLOYS AT 1350° F

By J. W. Freeman, E. E. Reynolds, and A. E. White
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SUMMARY

One of the objectives in the metallurgical research on heat-resisting metals for gas turbines has been the development of alloys which could operate at high temperatures under stresses similar to those permissible at room temperature. Data are presented in this report for alloys that come nearer to this objective at 1350° F than for any other alloys. Chromium-base alloys were tested which had rupture strengths as high as 73,000 and 54,500 psi, respectively, for fracture in 100 and 1000 hours. The highest similar values published for other alloys are in the order of 50,000 and 40,000 psi.

The chromium-base alloys have certain rather severe limitations in their present state of development. The most serious are brittleness at room temperature and the necessity for melting and casting under high vacuum to avoid the detrimental effects from the oxygen and nitrogen in air. Considerable progress has been made in overcoming these difficulties, and further improvement seems quite possible.

At the present time the most promising chromium-base alloy at 1350° F for buckets for gas turbines appears to be 60Cr-25Fe-15Mo with less than 0.05 percent carbon and from 0.5 to 0.7 percent silicon. This alloy can be machined and fabricated, with care, in the normal manner, and techniques have been worked out for casting buckets. The rupture strengths of this particular analysis are not the highest possible from chromium-base alloys. The higher molybdenum alloys have higher strength but have inferior engineering characteristics at room temperature.

INTRODUCTION

In the various research programs on alloys for gas turbines conducted in this country during the last 5 years, a primary objective has been the discovery of an alloy with sufficient resistance to the weakening effect of heat to permit operating stresses of the same order possible at room temperature. It seemed reasonably certain that none of the alloy systems available at the start of these programs could be modified to meet this requirement. Consequently, a number of studies were initiated to develop alloys based on an entirely new element. One of the most promising alloy systems, from both theoretical and practical considerations, involved the metal chromium as a base.

Alloys of this type were made available after a well-executed research program by the Climax Molybdenum Company under sponsorship of the OSRD (Project NRC-8). Certain chromium-base alloys were found to have exceptionally high rupture strength at 1600° F, although they were notably lacking in some other engineering properties such as toughness at room temperature. Consideration of the characteristics of these alloys together with the fact that most current gas turbines involved operation at metal temperatures below 1500° F indicated that it would be desirable to determine whether certain of the alloys might be outstanding at 1350° F.

The alloys included were to be selected jointly by representatives of the Climax Molybdenum Company and the University of Michigan. The alloy considered most promising contained 60 percent chromium, 25 percent iron, and 15 percent molybdenum. This particular analysis was selected because it was probably the strongest alloy with adequate properties for fabrication at room temperature. Adequate fabrication characteristics meant that the alloy was machinable and could be handled, with sufficient care, with a reasonable certainty that buckets could be made and installed in a gas turbine. It was recognized that other alloys containing more molybdenum probably would be stronger, but were considered too brittle for possible bucket use with current wheel fabricating techniques. Survey tests were made on other combinations of these elements within the range of machinable alloys in the system.

The rupture tests were performed at the University of Michigan under the sponsorship and with the financial assistance of the National Advisory Committee for Aeronautics. The casting and machining of suitable test specimens was performed by the Climax Molybdenum Company under the auspices of Project NRC-8 of OSRD.

TEST MATERIALS

The chemical composition of the alloys studied are given in table I as reported by the alloy producer. The five following basic compositions were included:

60Cr-30Fe-10Mo

65Cr-25Fe-10Mo

50Cr-35Fe-15Mo

60Cr-25Fe-15Mo

55Cr-25Fe-20Mo

Most of the work was concentrated on seven heats of the 60Cr-25Fe-15Mo alloy with carbon and silicon contents as variables. Two heats of 60Cr-30Fe-10Mo and only one heat each of the other alloys were considered.

The alloys were prepared in the Climax Molybdenum laboratory. Small experimental heats were vacuum melted and cast into bars of approximately specimen size. These bars were machined to the final size and furnished as 0.160-inch-diameter specimens with a 1-inch gage length.

All the heats were tested in the as-cast form. Specimens of four heats of the 60Cr-25Fe-15Mo alloy were prepared to show the effect of aging treatments on the properties of this alloy. The aging treatments were either 90 hours at 1600° F or 50 hours at 1800° F in a vacuum furnace as shown in table I.

PROCEDURE

The investigation was limited in scope to the evaluation of the 1350° F rupture properties of the five chromium-base alloys on the basis of only a very few tests on each alloy. If the properties of an alloy did not appear promising, no further tests were run as in the case of the 60Cr-30Fe-10Mo alloys. On other of the alloys, specimens sufficient for only one or two rupture tests were available; therefore, in most cases only two points were obtained for the curve of stress against rupture time.

Single-specimen-type rupture units, applying stress to the specimen by a simple beam through a system of knife edges, were used. The specimens were held at the test temperature for approximately 24 hours before applying the load, during which time the temperature distribution over the specimen was adjusted. Time-elongation data were obtained by measuring the drop of the beam during the test.

Because of the extreme room-temperature brittleness of some of the specimens, great care had to be exercised in placing the specimen into the rupture-test unit. Ordinary gage marks could not be used and total elongations were measured between the shoulders of the specimens. The high strength of the test materials and the comparatively low strength of available adapter materials caused trouble in successfully completing the tests.

Samples of the original casting and the completed rupture test specimens were examined metallographically. Photomicrographs at 100 and 1000 magnifications were taken of some of the materials. Electrolytic chromic acid was used as an etchant on all the metallographic specimens.

Vickers hardness tests were made on the metallographic samples of the original material and the completed rupture test specimens.

RESULTS

The characteristics of the alloys in the rupture test at 1350° F are shown by the data in tables I and II and by the usual log-log plots of stress against rupture time in figure 1. Comparative data for the cobalt-base alloy 422-19 are included in table II and figure 2. Fairly complete rupture-time curves were obtained for the strongest two alloys, 55Cr-25Fe-20Mo and 60Cr-25Fe-15Mo. The comparative values of rupture strength and ductility for the other alloys were based on extrapolations which are believed to be fairly reliable.

There was a distinct strength difference between the five groups of alloys. The 100-hour rupture strengths ranged from 43,000 psi for the 60Cr-30Fe-10Mo alloy up to 73,000 psi for the 55Cr-25Fe-20Mo alloy; 1000-hour rupture strengths ranged up to 54,500 psi for the strongest alloy, 55Cr-25Fe-20Mo. The low-carbon and high-silicon 60Cr-25Fe-15Mo had the highest strength of the various modifications of this alloy and had properties almost as high as the 55Cr-25Fe-20Mo alloy.

The ductility of the chromium-base alloys in the rupture tests was good. For the lower strength alloys elongations ranged from 10 to 48 percent; while it was not so high for the higher strength alloys, ranging from 4 to 19 percent.

The tests on the heat-treated specimens were not conclusive. Premature fracture occurred in the threaded ends of the specimens in all but one case. The one successful test, specimen 655-2HT of the 60Cr-25Fe-15Mo analysis, indicated that the heat treatment slightly lowered rupture strength.

The time-elongation curves for the tests showed either decreasing creep rates over the duration of the tests, or a period of decreasing rate followed by a constant rate until fracture occurred. The curves for the strongest two alloys are shown in figures 3 and 4 as typical examples of the deformation characteristics of the alloys. The disagreement between the total elongation values from the strain readings and measurements after fracture is due to two possible causes. The total elongation after fracture could be in error by 2 or 3 percent because of inherent inaccuracy of making such measurements between shoulders without gage marks. Considerable error could have been introduced in the time-elongation data through creep in the adapter system.

The alloys containing 15 and 20 percent molybdenum developed extremely high hardness during testing. The data in table I show values for these alloys as high as 600 or 700 Vickers Brinell. The alloys containing 10 percent molybdenum did not harden nearly so much.

Photomicrographs of the original material and the longer time rupture specimens of each group of alloys are shown in figures 5 through 9. All the original materials had similar solid-solution structures with some excess constituent in the grain boundaries. Precipitation had occurred in all the rupture specimens, although there was a marked degree of difference in the amount of precipitate in the different alloys. The time of the test was an important factor in that the amount of precipitate probably increased with testing time. It appears, however, that the alloys with 10 percent molybdenum did not tend to precipitate to so marked a degree as the higher molybdenum alloys. Increasing the carbon and silicon content of the 60Cr-25Fe-15Mo alloy resulted in more precipitated phase during rupture testing. The 55Cr-25Fe-20Mo alloy, the strongest of the materials tested, had a precipitate which appeared to be finer and seemed to occur along definite planes in the grains as stringers in contrast with the spheroidal type of precipitate occurring in the other alloys.

Only one heat-treated specimen was examined metallographically. The 90 hours at 1600° F resulted in slightly larger particles of the precipitated constituent in the 655-2HT specimen than in the as-cast 655 specimen.

DISCUSSION OF RESULTS

The rupture strengths, indicated by the data from this investigation, for the 55Cr-25Fe-20Mo and the 60Cr-25Fe-15Mo alloys, are much higher than published values for any other alloy at 1350° F. The rupture strengths in table II for alloy 422-19 were the highest known at the time this work was started. The 47,000-psi stress for rupture in 100 hours for alloy 422-19 is to be contrasted with an estimated value of 73,000 psi for the 55Cr-25Fe-20Mo alloy. The comparative curves of stress against rupture time for the cobalt- and chromium-base alloys in figure 2 show that the high strength of the latter is maintained for long time periods.

These high rupture strengths for the chromium-base alloys should be considered primarily as suitable proof that the further development of these alloys is justified. The alloys used in this investigation are certainly not the strongest known in the system. Data at 1600° F (see references 1 and 2) indicated considerably higher strengths for a 60Cr-15Fe-25Mo alloy. Alloys with more than 20 percent molybdenum were not included in this investigation, however, because of their brittleness at room temperature and the necessity to grind rather than machine.

The production of even the 15-percent-molybdenum alloys at the present time is little more than a laboratory art in spite of the tremendous advances of the last few years. The Climax Molybdenum Company did demonstrate, however, that it was feasible to cast buckets and prepare them for installation in a turbine rotor. The major difficulty in melting and casting is that only very small heats can be made because of the extremely high vacuum needed for deoxidation and for prevention of contamination from nitrogen in air. Considerable care must be exercised in handling the alloys at room temperature. Even though they can be machined, they have little resistance to shock loads and stress concentrations. The success of the Climax Molybdenum Company in their efforts to reduce these drawbacks to the alloys should be an encouragement to further development of the alloys.

At present, the 10-percent-molybdenum chromium-base alloys are not particularly promising for gas-turbine service. Their rupture strengths are no better than some of the cobalt-base alloys which are much easier to produce.

The data on the effect of carbon and silicon on the 60Cr-25Fe-15Mo alloys suggest that the properties of these alloys in general may be subject to modification and improvement by the addition of other elements. In the alloys studied, the indications are that the best properties at 1350° F are obtained with carbon contents of the order of 0.02 percent and silicon contents of 0.7 percent. Alloys with carbon contents of 0.10 percent, with or without high silicon, are not quite so good but better than low-carbon, low-silicon alloys. Unfortunately, no data are available regarding the effect of such composition modifications on the room-temperature characteristics. No data have been obtained to indicate what the effect of carbon and silicon modifications might be on the properties of the 20-percent-molybdenum alloys.

All the alloys considered were subject to strong precipitation reactions. This cannot be attributed to the formation of carbides. The change in hardness of 60Cr-25Fe-15Mo alloys was nearly equal for 0.01- and 0.10-percent-carbon heats. The nitrogen content of these alloys was very low initially. The precipitating constituent is therefore an intermetallic compound. In their intensive study of the alloys the Climax Molybdenum Company were not able to identify the compound. They concluded (see references 1 and 2) that it did not have either a cubic or a hexagonal structure and therefore was not hexagonal Fe_3Mo_2 or FeMo, but might be an iron-chromium compound. The increasing amount of precipitation after rupture testing in the samples with increasing molybdenum content indicates that the compound is related to molybdenum in some manner. The amount of precipitate in the 15-percent-molybdenum alloys also appeared to increase with iron content.

The metallurgy of these alloys is not well understood. It appears certain, however, that the high strength at 1350° F is due primarily to the precipitation characteristics. The matrix material may be stronger than in other alloys, although the data on low-molybdenum alloys do not bear this out. This precipitation develops high hardness after exposure at 1350° F and extreme brittleness at room temperature. The cause for the initial brittleness of as-cast specimens at room temperature is not clear because the microstructure does not indicate the precipitation reaction to be the cause. It may be related to a similar, but less severe, phenomenon encountered in some iron-chromium alloys.

The retention of most of the strength in the one alloy tested after aging for 90 hours at 1600° F indicates that the strengthening effect from the precipitation reaction would be stable at 1350° F. This is verified by the curves of stress against rupture time. The slope of the curves is not very steep, whereas unstable materials usually have a steep slope. In fact, the curves, as shown by figure 2, are parallel to those for the cast cobalt-base alloy.

CONCLUSIONS

The very high strength exhibited by the 55Cr-25Fe-20Mo and 60Cr-25Fe-15Mo alloys in rupture tests at 1350° F indicates that the chromium-base alloys are potentially among the most promising known for bucket service in gas turbines. These alloys both had rupture strengths considerably above those of any previously known alloy with adequate ductility at 1350° F. A strong precipitation reaction occurs at 1350° F which is stable at 1350° F and is probably responsible for the high strength.

The chromium-base alloys have several limitations at their present state of development. The most serious are:

1. Very low shock resistance and sensitivity to stress concentrations at room temperature. This is increased by the precipitation reaction during rupture testing.

2. The alloys must be melted and cast under high vacuum in order to avoid the detrimental effects of oxygen and nitrogen. For this reason their production is little more than a laboratory art at the present time.

In the development of the alloys, a great deal of progress was made in reducing these shortcomings. The last alloys made were far superior to the early alloys in room-temperature properties. Equipment and techniques have been developed which can be used to produce turbine buckets. There seems to be every reason to expect that a great deal of further improvement would be possible.

At the present time the alloy most promising for service at 1350° F appears to be a 60Cr-25Fe-15Mo alloy with less than 0.05 percent carbon and 0.5 to 0.7 percent silicon. This alloy has the best combination of strength at 1350° F and engineering properties at room temperature. It can be machined and processed in the chill-cast condition, with care, in the normal manner. Higher strengths can be obtained from alloys with higher molybdenum contents, but at the present time their inferior properties at room temperature offset their higher strength.

University of Michigan,
Ann Arbor, Mich., August 19, 1946.

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2. Parke, R. M., and Bens, F. P.: Chromium-Base Alloys. Symposium on Materials for Gas Turbines. A.S.T.M., June 1946.

3. Freeman, J. W., Rote, F. B., and White, A. E.: The High Temperature Characteristics of 17 Alloys at 1200° and 1350° F. NACA ACR No. 4C22, March 1944. (Unclassified)

TABLE I

RUPTURE TEST RESULTS AT 1350° F ON CHROMIUM-BASE ALLOYS

Specimen number	Chemical analysis C	Si	Cr	Fe	Mo	Condition	Stress (psi)	Rupture test results: Time for rupture (hr)	Elongation (percent)	Reduction of area (percent)	Vickers hardness: Original	after testing
608	0.03	0.14	61.96	29.14	8.73	As cast	50,000	38	40	35	402	434
530-1	.04	.03	59.14	30.97	9.82	As cast	50,000	39	48	44	420	474
530-2							(not tested)					
678-1	.03	.19	65.49	24.62	9.67	As cast	50,000	113	13	13	408	434
678-2							45,000	191	19.5	17		462
534-1	.02	.11	49.26	35.93	14.68	As cast	50,000	270.5	10	13.5	395	670
534-2							55,000	60	16	23		660
522	.05	.17	58.60	25.71	15.47	As cast	45,000	(overheated and broke at 450 hr)[a]			450	-------
612-2	.01	.13	58.86	25.11	15.89	As cast	50,000	484	9	9	432	628
655-1	.02	.19	59.80	25.23	14.76	As cast	50,000	530	19	17	428	604
655-2HT						90 hr at 1600° F	50,000	434	10	12		580
655-3HT						50 hr at 1800° F	50,000	(broke in threads during loading)				-------
657-1	.08	.19	60.70	24.22	14.81	As cast	55,000	347	11	11	410	592
657-2HT						90 hr at 1600° F	55,000	(broke in threads during loading)				-------
554-2	.04	.57	59.43	24.96	15.00	As cast	50,000	1414	8	6.2		663
554-3HT						Aged	55,000	(broke in threads during loading)				-------
679-1	.02	.70	60.44	24.21	14.63	As cast	63,800	(broke in threads at 182 hr)[a]			442	-------
679-2							55,000	611	5	6		677
679-3							70,000	76	4	6		642
634-1	.10	.70	59.09	24.55	15.56	As cast	50,000	985	5	5	466	670
634-2HT						90 hr at 1600° F	50,000	(broke in threads at 464 hr)[a]				-------
537-1	.03	.06	54.56	24.83	20.52	As cast	50,000	(broke in threads at 2040 hr)[a]			458	748
537-2							60,000	468	6	5		716

[a]Ductility of specimens which overheated or broke in threads:

Specimen	Elongation (percent)	Reduction of area (percent)
522	9	9
679-1	5	10
534-2HT	4	4
537-1	3	2.5

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TABLE II

RUPTURE PROPERTIES AT 1350° F OF CAST CHROMIUM-BASE ALLOYS
AND COMPARATIVE PROPERTIES OF 422-19 ALLOY

Alloy	C	Si	Stress for rupture in indicated time periods (psi)			Estimated elongation to rupture (percent)		
			100-hour	500-hour	1000-hour	100-hour	500-hour	1000-hour
60Cr-30Fe-10Mo	0.03 .04	0.14 .03	43,000[a]	---------	---------	40	---------	---------
65Cr-25Fe-10Mo	.03	.19	51,000	36,500[a]	---------	13	19[a]	---------
50Cr-35Fe-15Mo	.02	.11	53,000	48,000	---------	16	10	---------
60Cr-25Fe-15Mo	.01	.13	---------	50,000[a]	---------	---------	9	---------
	.02	.19	---------	50,500[a]	---------	---------	19	---------
	.08	.19	---------	52,500[a]	---------	---------	10	---------
	.04 .02	.57 .70	68,000	56,500	52,000	4	5	5
	.10	.70	---------	---------	50,000[a]	---------	---------	5
55Cr-25Fe-20Mo	.03	.06	73,000[a]	59,500	54,500	---------	6	5
422-19[b] (25Cr-15Ni-52Co-6Mo)	.40	.51	47,000	39,500	36,000	30	15	10

[a]Estimated from incomplete data.
[b]See reference 3.

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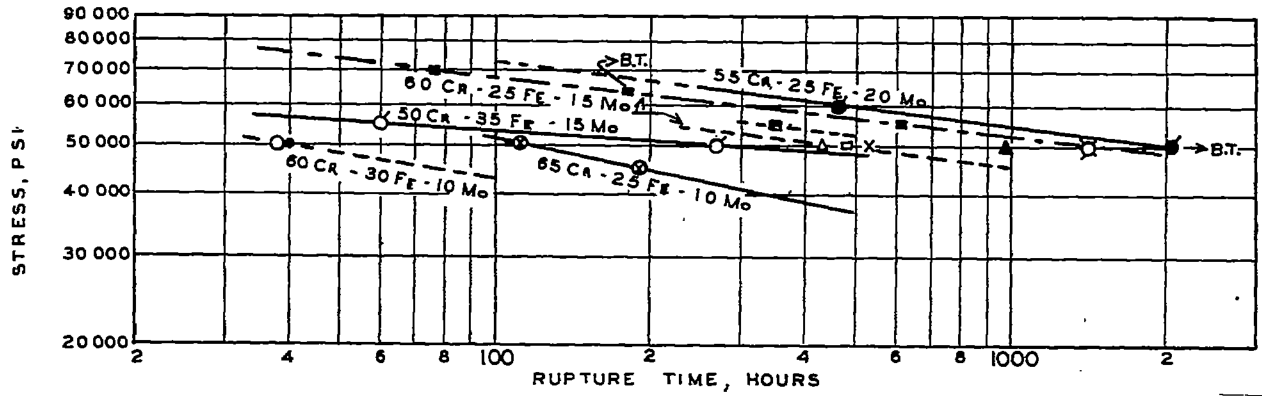
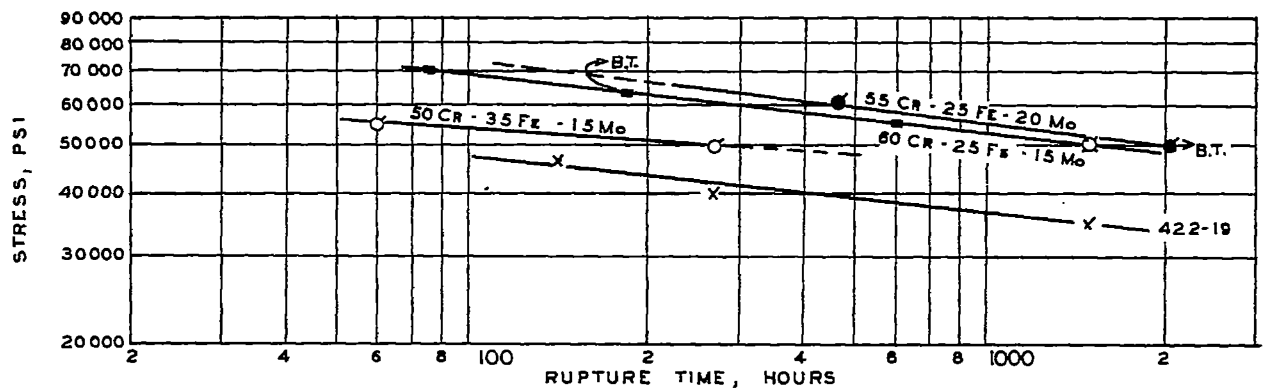


SYMBOL	HEAT NUMBER	CODE ALLOY CR	FE	Mo	C	SI	CONDITION
○	608	60	30	10	0.03	0.14	
●	530				.04	.03	
⊗	678	65	25	10	.03	.19	AS CAST
♂	534	50	35	15	.02	.11	
□	612	60	25	15	.01	.13	
×	655				.02	.19	
△	655-2HT				.02	.19	90 HR AT 1600° F
▪	657				.08	.19	
¤	554				.04	.57	
■	679				.02	.70	AS CAST
▲	634				.10	.70	
♂	537	55	25	20	.03	.06	

B.T. = SPECIMEN BROKE IN THREADED SECTION

FIGURE 1.- STRESS-RUPTURE TIME CURVES AT 1350° F FOR CAST CHROMIUM-BASE ALLOYS.



B.T. = SPECIMEN BROKE IN THREADED SECTION

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FIGURE 2.- COMPARATIVE STRESS-RUPTURE TIME CURVES AT 1350° F FOR CAST CHROMIUM- AND COBALT-BASE ALLOYS.

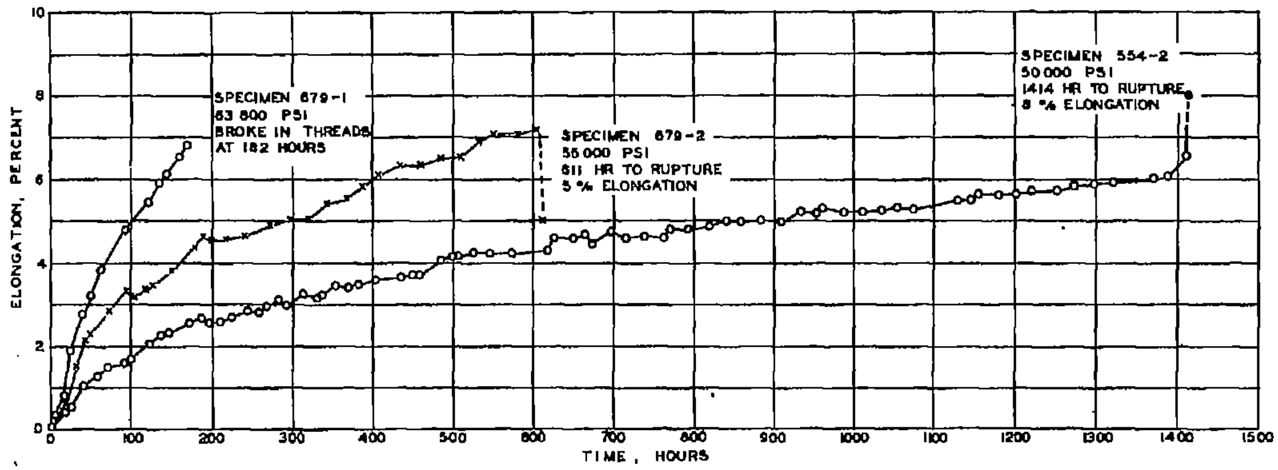

FIGURE 3.- TIME-ELONGATION CURVES FOR RUPTURE TESTS AT 1350° F ON CAST 60 Cr-25 Fe-15 Mo ALLOYS. HEATS 554 AND 679

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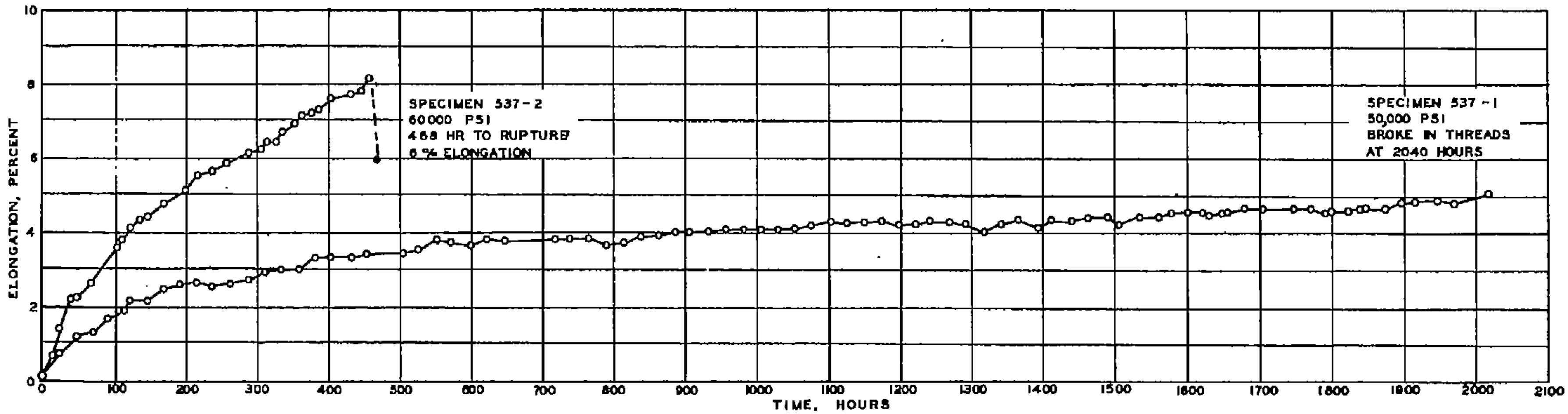

FIGURE 4.- TIME-ELONGATION CURVES FOR RUPTURE TESTS AT 1350° F ON CAST 55 Cr - 25 Fe - 20 Mo ALLOY. HEAT 537

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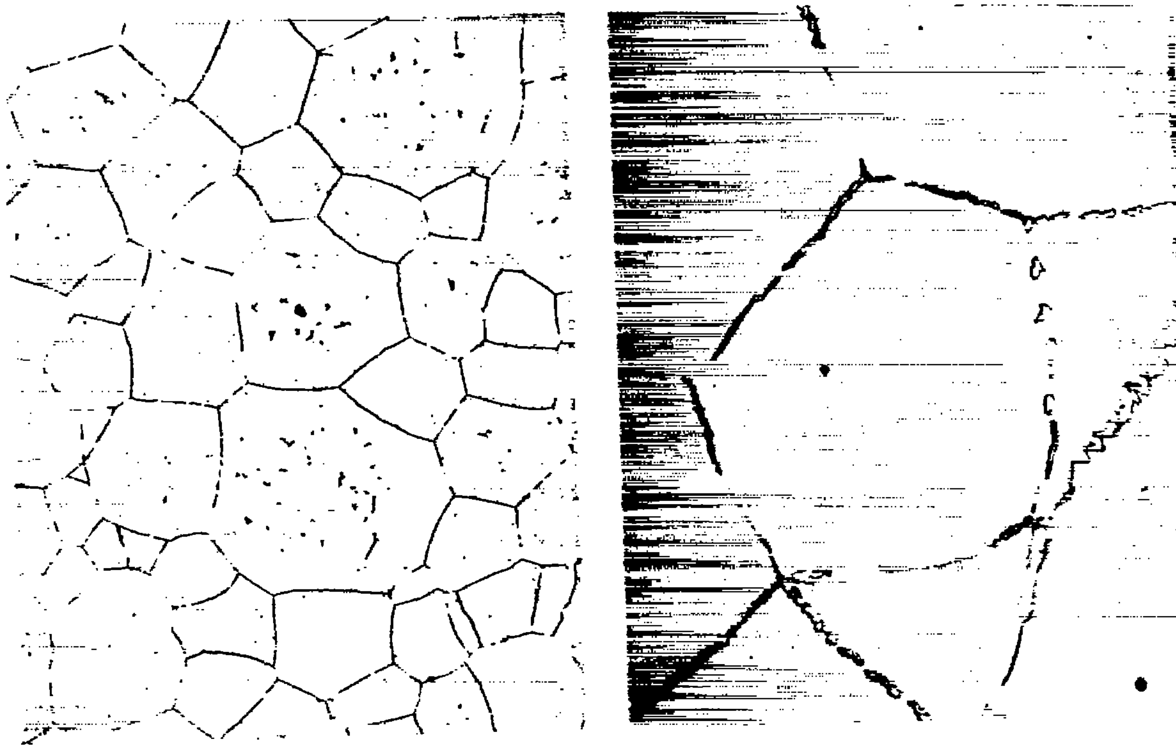
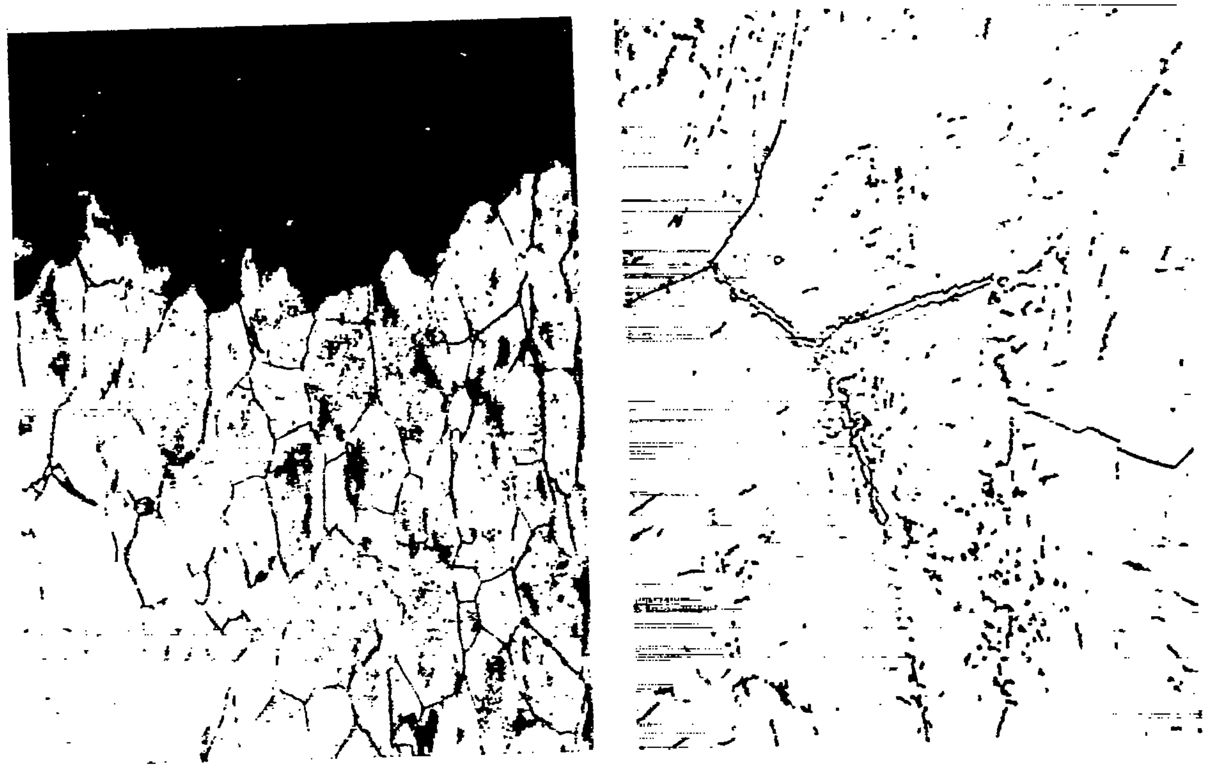


100X (a) 1000X

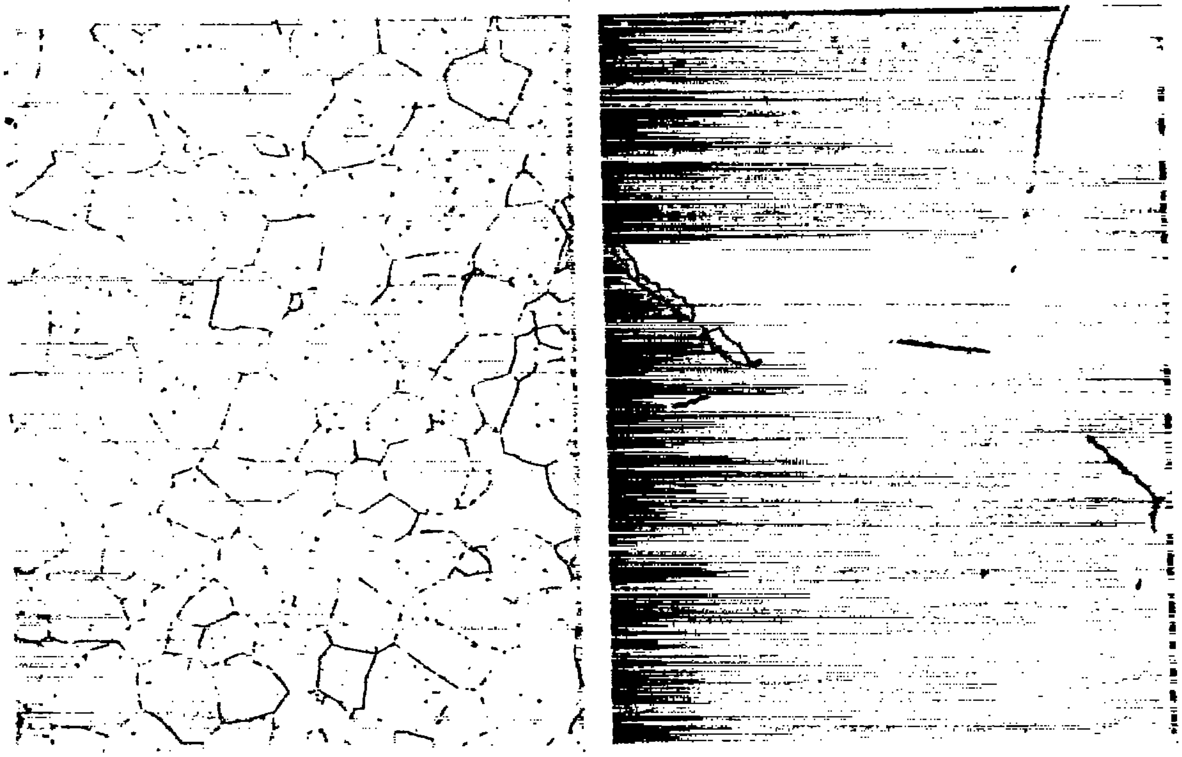
Original microstructure of heat 530: 0.04C, 0.03Si - As cast.



Fracture - 100X (b) Interior - 1000X

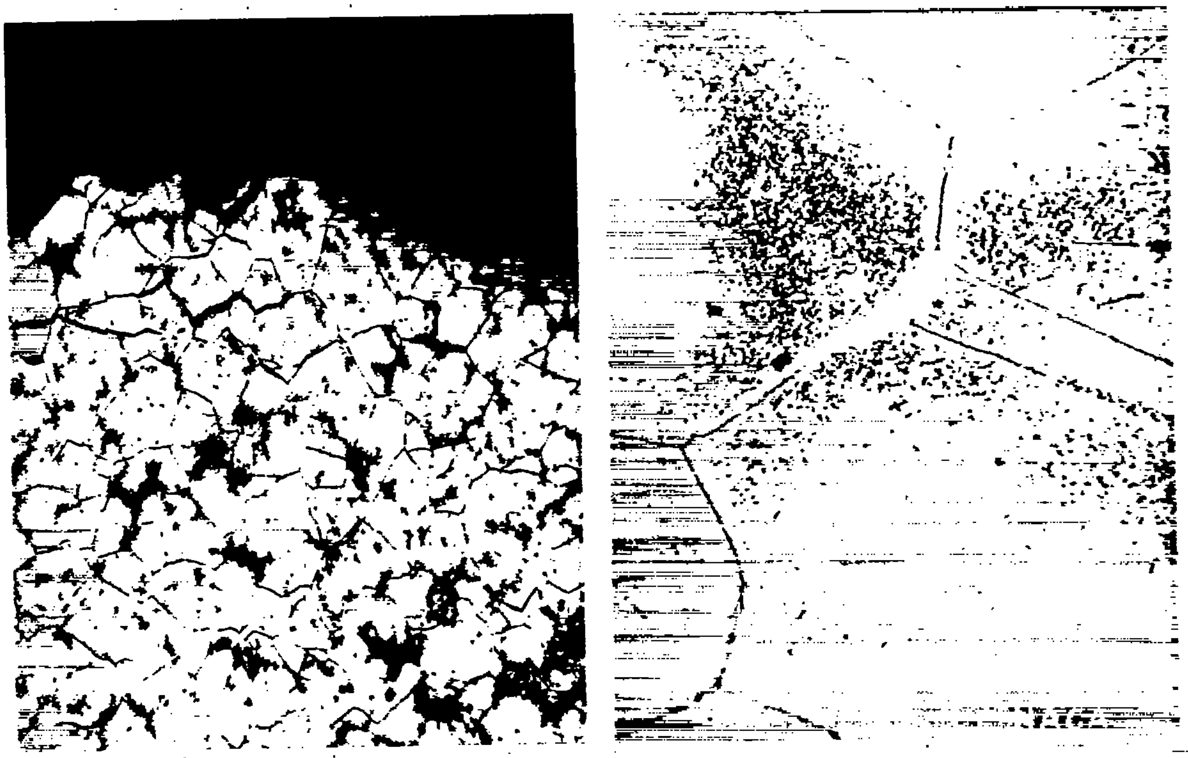
Rupture specimen 530-1: 39 hours for rupture at 1350° F under 50,000 psi.

FIGURE 5.- MICROSTRUCTURES OF 60Cr-30Fe-10Mo ALLOY.



100X (a) 1000X

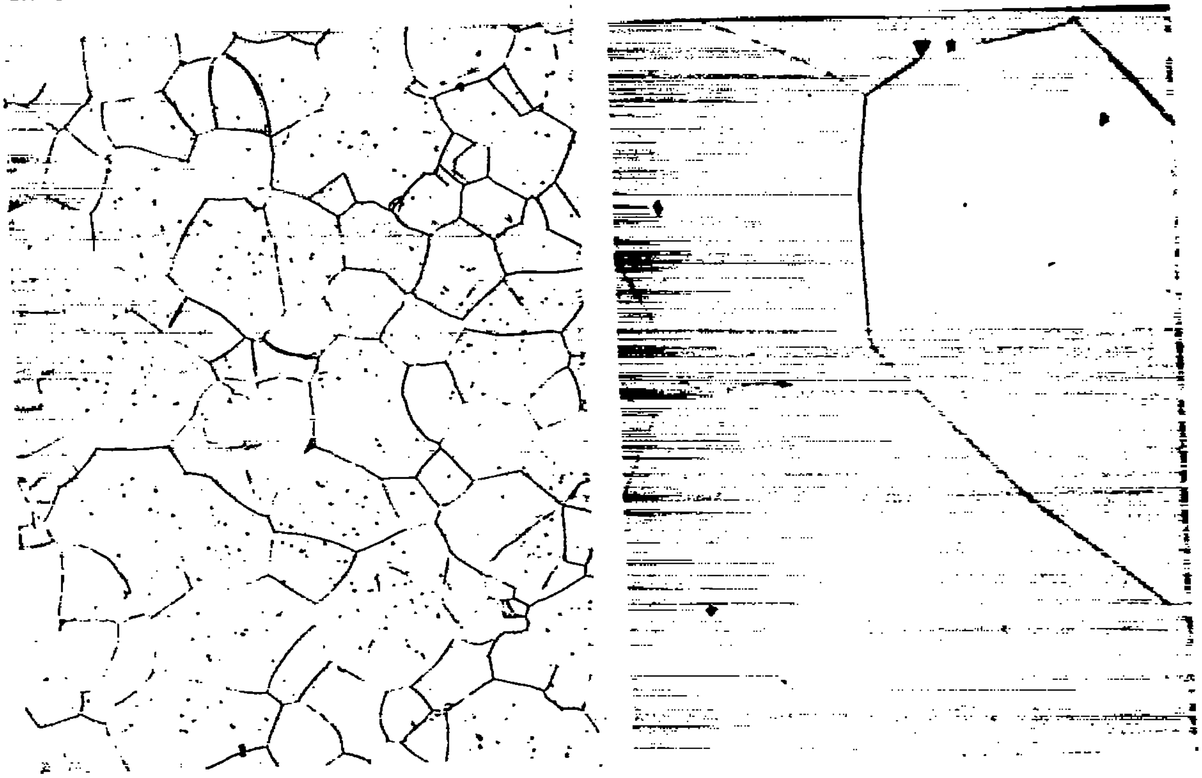
Original microstructure of heat 678: 0.03C, 0.19Si - As cast.



Fracture - 100X (b) Interior - 1000X

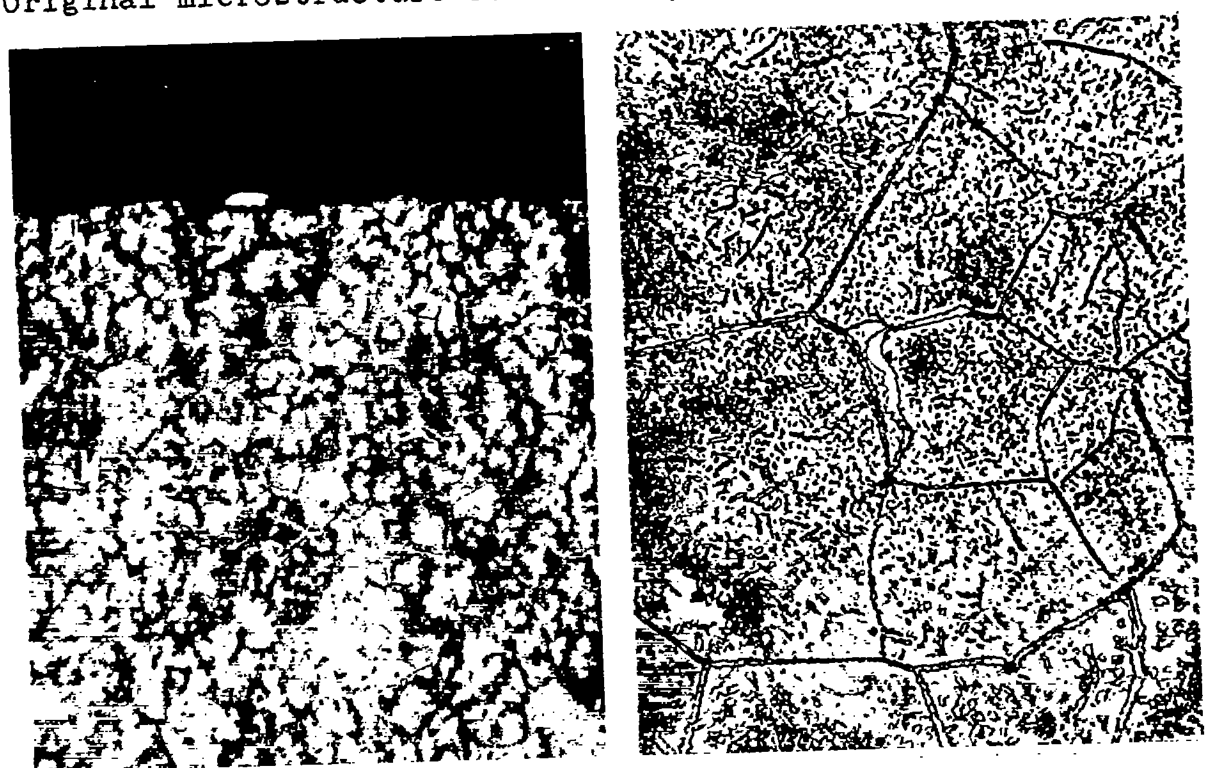
Rupture specimen 678-2: 191 hours for rupture at 1350° F under 45,000 psi.

FIGURE 6.- MICROSTRUCTURES OF 65Cr-25Fe-10Mo ALLOY.



100X (a) 1000X

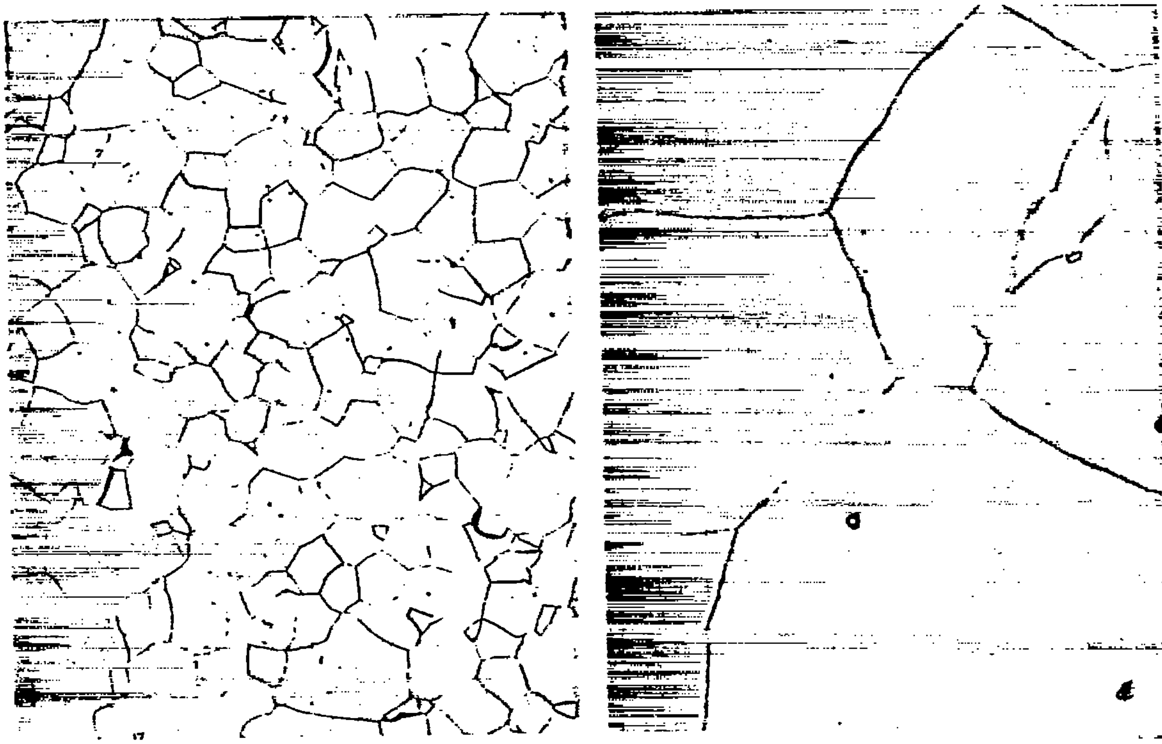
Original microstructure of heat 534: 0.02C, 0.11Si - As cast.



Fracture - 100X (b) Interior - 1000X

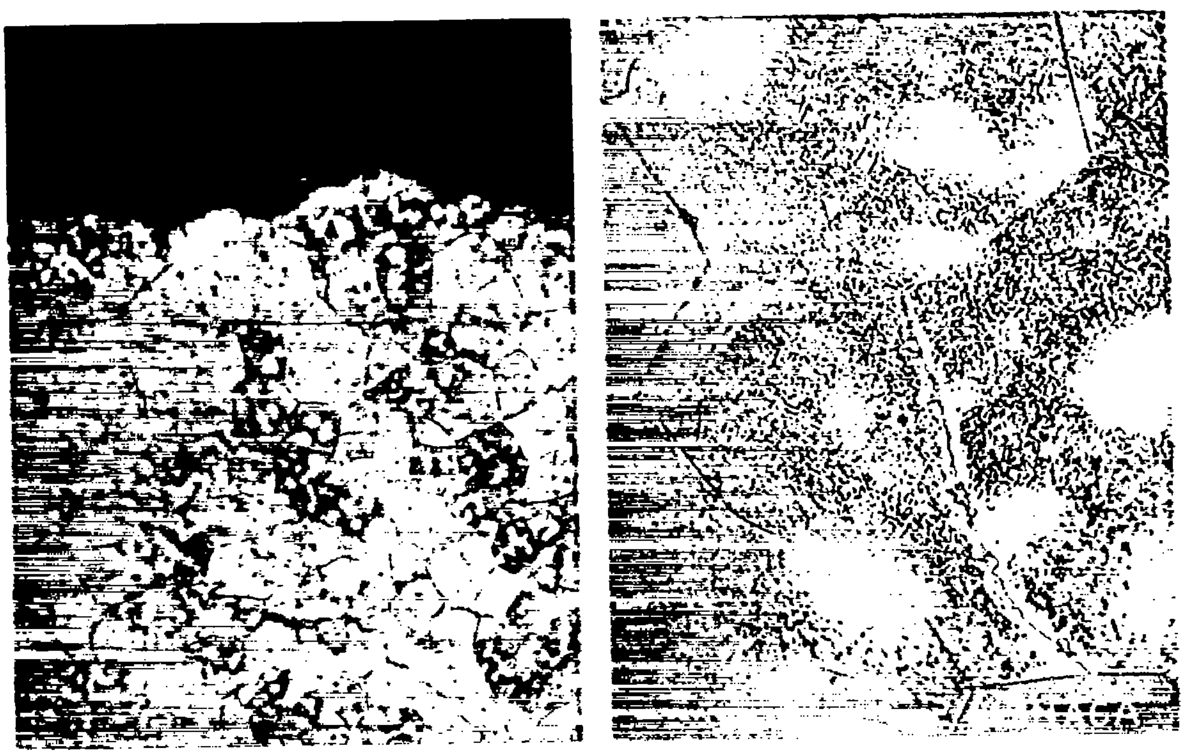
Rupture specimen 534-1: 270.5 hours for rupture at 1350° F under 50,000 psi.

FIGURE 7.- MICROSTRUCTURES OF 50Cr-35Fe-15Mo ALLOY.



100X (a) 1000X

Original microstructure of heat 679: 0.02C, 0.70Si - As cast.



Fracture - 100X (b) Interior - 1000X

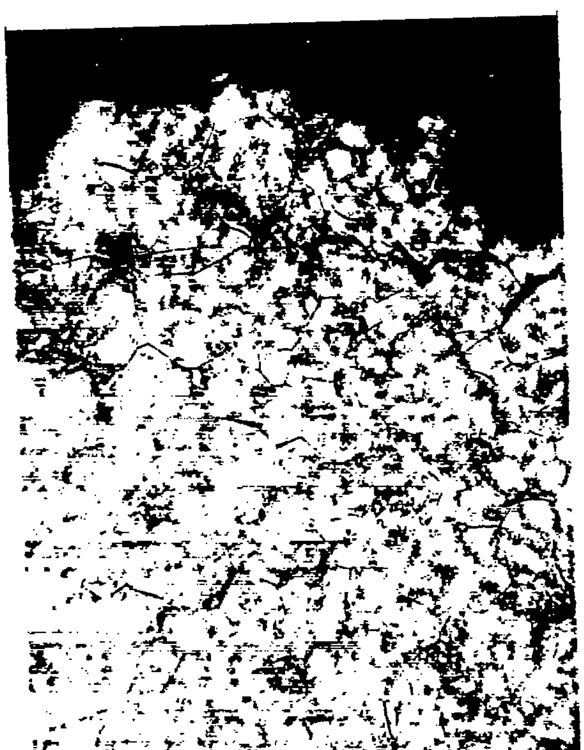
Rupture specimen 679-2: 611 hours for rupture at 1350° F under 55,000 psi.

Figure 8.- MICROSTRUCTURES OF 60Cr-25Fe-15Mo ALLOYS.

(c)

Fracture - 100X Interior - 1000X

Rupture specimen 655-1: 0.02C, 0.19Si - As cast: 530 hours for rupture at 1350° F under 50,000 psi.

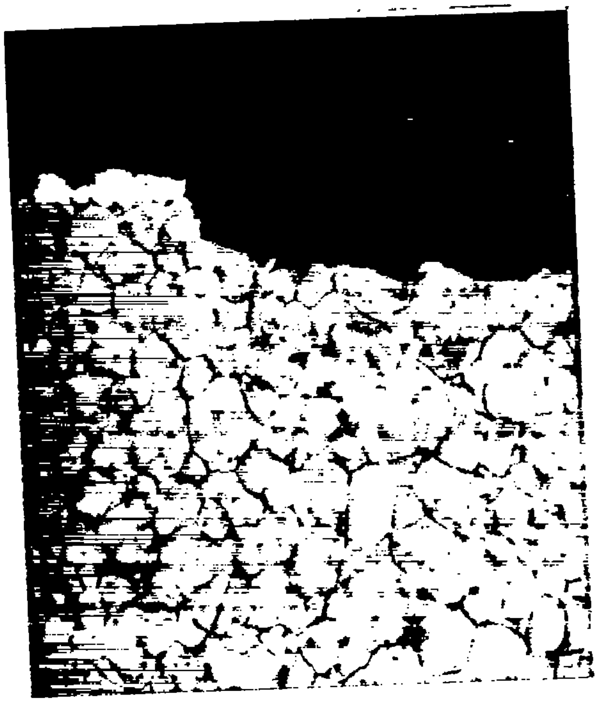


(d)

Fracture - 100X Interior - 1000X

Rupture specimen 655-2HT: 0.02C, 0.19Si - Cast + 90 hours at 1600° F: 434 hours for rupture at 1350° F under 50,000 psi.

Figure 8.- Continued.



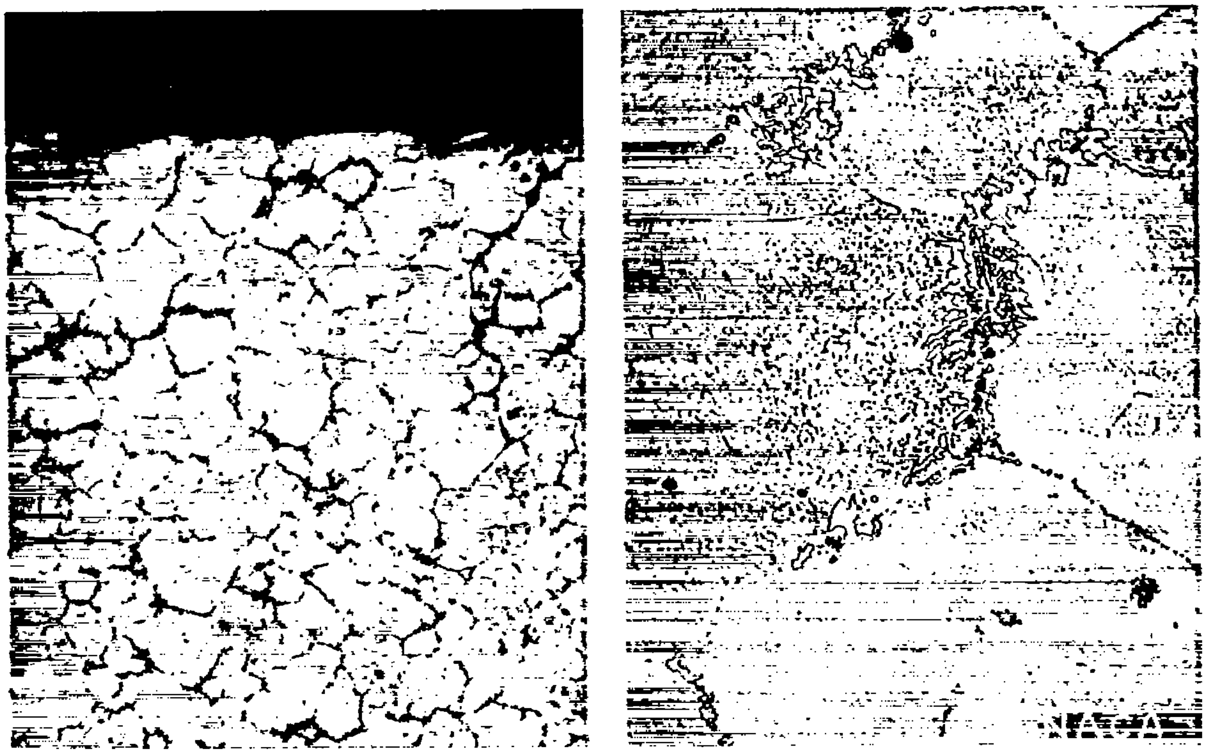
Fracture - 100X (e) Interior - 1000X

Rupture specimen 657-1: 0.08C, 0.19Si - As cast:
347 hours for rupture at 1350° F under 55,000 psi.

Fracture - 100X (f) Interior - 1000X

Rupture specimen 554-2: 0.04C, 0.57Si - As cast:
1414 hours for rupture at 1350° F under 50,000 psi.

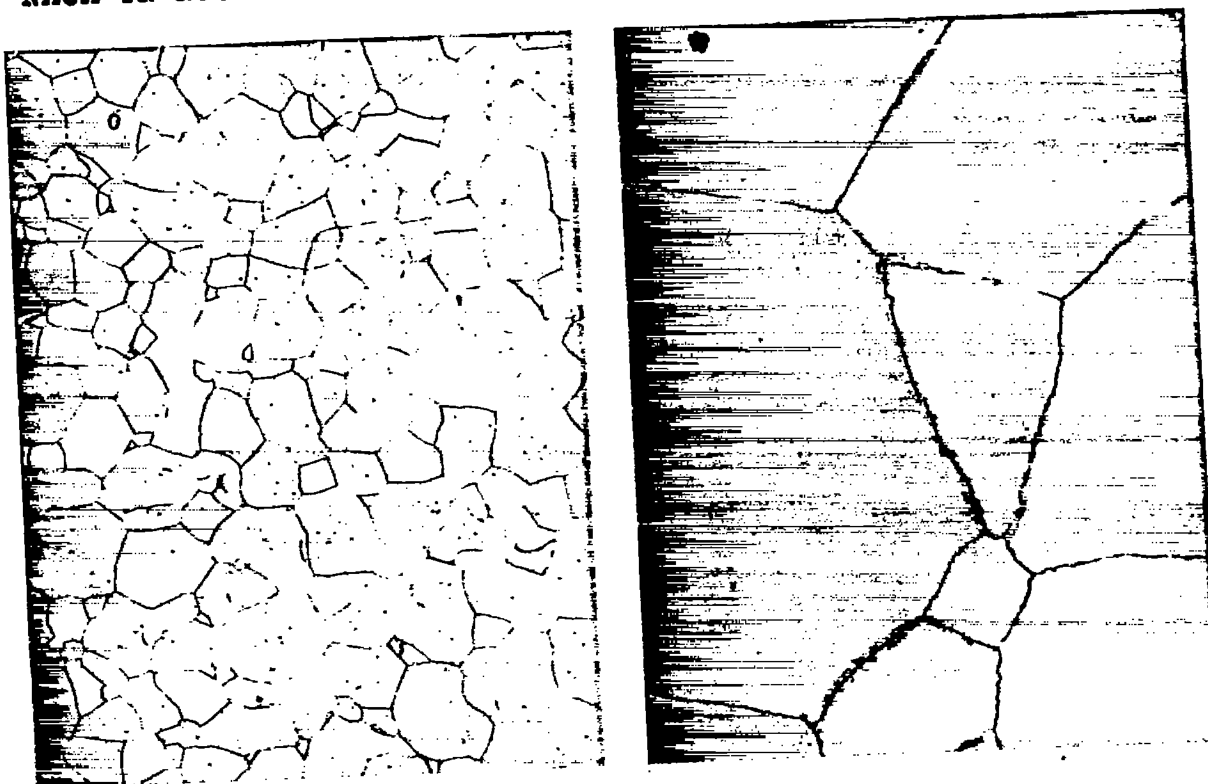
Figure 8.- Continued.



Fracture - 100X (g) Interior - 1000X

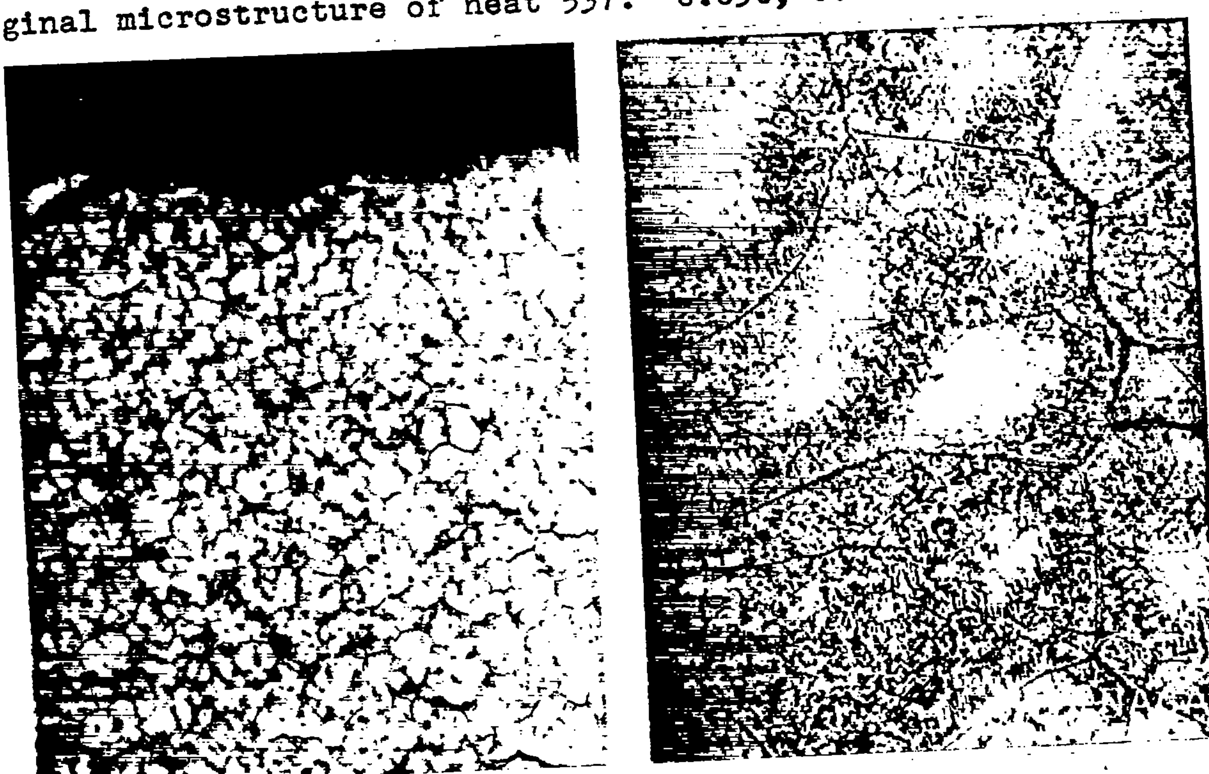
Rupture specimen 634-1: 0.10C, 0.70Si - As Cast: 985 hours for rupture at 1350° F under 50,000 psi.

Figure 8.- Concluded.



100X (a) 1000X

Original microstructure of heat 537: 0.03C, 0.06Si - As cast.



Fracture - 100X (b) Interior - 1000X

Rupture specimen 537-2: 468 hours for rupture at 1350° F under 60,000 psi.

FIGURE 9.- MICROSTRUCTURES OF 55Cr-25Fe-20Mo ALLOY.